

SOLUBILITY AND DIFFUSION STUDIES IN ALKALI METALS

SIXTH QUARTERLY REPORT

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

LEWIS RESEARCH CENTER

Under Contract No. NAS3-4163



ATOMICS INTERNATIONAL

a division of North American Aviation, Inc.

N 65-20886

(ACCESSION NUMBER)

27

(PAGES)

CR-54096

(NASA CR OR TMX OR AD NUMBER)

(THRU)

(CODE)

(CATEGORY)

QFST1

~~QFST1~~ PRICE(S) \$

Hard copy (HC)

Microfiche (MF)

\$2.00

\$0.50

**SOLUBILITY AND DIFFUSION STUDIES
OF ULTRA PURE TRANSITION ELEMENTS IN
ULTRA PURE ALKALI METALS

SIXTH QUARTERLY REPORT
(October 4, 1964 - January 2, 1965)**

**By
R.L. McKISSON
R.L. EICHELBERGER**

**Prepared for
National Aeronautics and Space Administration
Lewis Research Center**

**Technical Management
NASA - Lewis Research Center
R.A. Lindberg**

ATOMICS INTERNATIONAL

**A DIVISION OF NORTH AMERICAN AVIATION, INC.
P.O. BOX 309 CANOGA PARK, CALIFORNIA**

**CONTRACT: NAS3-4163
ISSUED: MARCH 5, 1965**

NOTICES

This report was prepared as an account of Government-sponsored work. Neither the United States nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA:

- A) Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately-owned rights; or
- B) Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method or process disclosed in this report.

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employee or contractor of NASA or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with NASA, or his employment with such contractor.

AVAILABILITY NOTICE

Qualified requestors may obtain copies of this report from:

National Aeronautics and Space Administration
Office of Scientific and Technical Information
Washington 25, D.C.
Attn: AFSS-A

TABLE OF CONTENTS

	Page
Title Page.....	i
Notices.....	ii
Table of Contents.....	iii
List of Figures.....	iv
List of Tables.....	iv
I. Introduction.....	1
II. Summary.....	2
III. Technical Program.....	3
Solubility Testing.....	3
Material Procurement, Preparation, and Characterization.....	9
Chemical Analysis.....	10
IV. Next Report Period Activities.....	19
V. Project Reports Issued on this Contract.....	20
VI. AI Internal Reports Issued on this Contract.....	21
VII. References.....	22

LIST OF FIGURES

	<u>Page</u>
1. Test Capsule Assembly.....	16
2. Cooling Curves for Quenching Using Chill Blocks.....	17
3. Concentration of Iron Dissolved in Potassium Containing 16 ppm O.....	18

LIST OF TABLES

1. Analyses of the First Batch of Potassium.....	12
2. Analyses of the Second Batch of Potassium.....	13
3. Summary of Experimental Data.....	14, 15

SOLUBILITY AND DIFFUSION STUDIES OF ULTRA
PURE TRANSITION ELEMENTS AND COMPOUNDS IN ULTRA PURE
ALKALI METALS

By R. L. McKisson and R. L. Eichelberger

I. INTRODUCTION

The purposes of this study are: 1) to define the solution process and determine the equilibrium solubility of highly purified transition metals and selected compounds in highly purified liquid alkali metals, and 2) to measure the diffusion of transition metals and constituents of selected compounds in highly purified liquid alkali metals. The material combinations which are to be considered for both the solution and diffusion studies are: iron, niobium, tantalum, molybdenum, tungsten, zirconium, rhenium, vanadium, hafnium, beryllium oxide, niobium monoxide, tantalum monoxide, zirconium dioxide, zirconium carbide, vanadium monoxide, oxygen-saturated zirconium, and oxygen-saturated hafnium with liquid potassium, and niobium and beryllium oxide with liquid lithium. Specific combinations from the above list will be selected, and it is expected that some combinations will not be investigated.

The studies to be made include the measurement of solubility in the alkali metal at temperatures up to 1200°C, coupled with the investigation of the rate-controlling step and its energy of activation; and the measurement of the liquid state diffusion rate and its energy of activation, also to 1200°C.

It is the goal of this study to develop solubility and diffusion data for well-characterized experimental systems, in which the number and range of complicating variables are minimized, in the hope that such data will not only further the understanding of these processes in alkali metal systems, but will also be of use in the materials selection and design of space electrical power system components.

II. SUMMARY

20886

Solubility testing has begun, and tests have been run on high-purity polycrystalline iron samples, and on high purity niobium and molybdenum samples. An experimental difficulty has become apparent in that the original slip-fit seal between the collector and the crucible is not adequate to prevent the transfer of potassium out of the crucible during testing. It is not yet known with certainty what the mechanism of transfer is, but there is evidence that the temperature differences imposed on the capsule during the quench operation may be the source of the trouble. During the quench the capsule wall is thought to cool rapidly and to become 30-40°C cooler than the potassium in the crucible-collector, so that vapor transfer from the inside of the crucible to the outside occurs. Techniques for effecting a diffusion bond seal between the collector and the crucible are being tested and evaluated. Tentative values of the solubility of very pure iron in potassium from the present series of tests are: 17 wppm at 800°C; and 100 wppm at 1000°C.

The results of the tests carried out thus far indicate the solubility in potassium of our ultra-high purity iron is substantially lower than that of Armco iron, but the temperature dependencies of solubility of the two sets of data appear to be nominally equal.

One excellent molybdenum single crystal crucible has been received and is being readied for testing. The crucibles not yet delivered are: one molybdenum; one niobium; and two tantalum.

The current batch of potassium shows metallic impurity levels lower than those of the normally available "high-purity standard" materials. The only metallic elements detected spectroscopically are: <1 ppm Ag, <1 ppm Mg, 10 ppm Na, and <5 ppm Si.

Author

III. TECHNICAL PROGRAM

Solubility Testing

Solubility testing was begun during this report period. Two trial runs were made using the first batch of product potassium, and the later runs were made using the second batch of potassium product. The preparation of the second batch of product potassium followed a cleaning operation on the extruder. The unit was disassembled and thoroughly cleaned to ensure that it would be free of contamination remaining from the first batch whose analysis is shown in Table 1. Following the cleaning and re-assembly, three batches of potassium were distilled and collected in the extruder. The first batch was ungettered, and was merely used to clean out the still, condenser and extruder barrel, and was discarded in its entirety. The second batch was gettered for 8 hours at 650°C and was also used to clean up the internal parts of the purification unit. Most of this batch was immediately discarded but a small amount (about 30 grams) was left to form a plug in the delivery tube of the extruder. The third batch of potassium was gettered for 8 hours at 650°C, and the first 40 gms were discarded, together with that remaining from the second batch. The middle fraction, about 200 grams, was then collected in the extruder to be the purified product. The analysis of this product is shown in Table 2.

The first trial solubility test run was made using a high purity polycrystalline iron crucible. The test was scheduled as an 8 hour run at 800°C using the potassium containing 40-70 ppm oxygen. The run (#311) was carried out even though the analyses of that batch of potassium showed unexplainably large differences in the oxygen content of presumably identical samples. The purpose of the run was to obtain added experience in operating the test equipment, and as an indirect means of evaluating the oxygen level.

The test was terminated after 2 hours and 35 minutes because a vacuum leak developed at the ball joint of one of the manipulators. As soon as possible after the leak developed and the power was turned off, the test capsule was moved into the chill block in order to salvage the sample. The

leak was quite large and developed suddenly so that the furnace heating element was very badly damaged, and the furnace radiation shields were also markedly affected. The surface of the test capsule was oxidized, but examination of the opened test capsule showed no evidence that the internal parts were affected. Because there seemed to be no adverse effects on the sample, the potassium was submitted for analysis.

The repair of the furnace required about two weeks and involved replacing the tantalum heating element, cleaning the radiation shields and the cup in which the test capsule seats, replacing the TZM shaft which supports the cup and capsule in the furnace, and repairing some of the thermocouple feed-through seals. Because of the untimely termination of the first run, and the desire to obtain additional operating experience, the second run (#314) was set up to run for 2 hours and 35 minutes at 800°C using potassium from the first product batch. A zirconium tab was loaded into the iron crucible with the potassium to investigate the effect of gettering on the amount of iron dissolved. The results of these two runs are summarized in Table 3, which shows 25 wppm iron to have dissolved in run #311, in which all of the potassium solvent was transferred into the collector. The iron concentration in the solvent collected in run #314 is seen to be 15 wppm, but only 76% of the potassium was found in the collector. If the iron found in the collector is considered to have been dissolved by the total amount of potassium, the concentration becomes 11.4 wppm iron.

The observed reduction in the amount of iron dissolved in run #314 suggests that the oxygen concentration in the potassium solvent was probably high, as is indicated in Table 1. This conclusion stems from observations reported from other laboratories that the presence of a getter markedly reduces metal solubility in systems having high oxygen content in the liquid metal.⁽¹⁾ If the oxygen content is below about 20 wppm, the effect of the gettering action is small. Since the present difference is about a factor of two, it can be concluded that the solvent potassium probably does have >40 ppm oxygen, as indicated by the mercury amalgamation analysis.

The problem of potassium transfer first noticed in run #314 has appeared in many of our tests, as is indicated in Table 3. The transfer of potassium out of the crucible is completely undesirable, and some means of preventing it must be found. A sketch of the assembly is shown in Figure 1. Normally, one would expect a small amount of potassium to be found between the crucible and the capsule, because potassium vapor will fill all of the open space within the capsule during testing. Upon cooling at the end of a run, this vapor will condense to leave a small amount of potassium (~ 0.1 gm) outside the crucible. Much more has been frequently found. An analysis of the process lends to three possible mechanisms by which massive amounts of potassium might transfer out of the crucible-collector sub-assembly. First, there may be spillage of the potassium as the capsule is inverted and the metal flows into the collector. The joint between the two parts is not a tight joint and certainly some material could run out. However, tests using acetone and water as fluids indicate that only a few drops will spill under the worst conditions if the collector is properly seated in the originally designed crucible lip. One would not expect losses of a half gram or more from this source if the collector mouth is maintained in position in the crucible lip.

The second mechanism considered is the evaporation-condensation transfer of the potassium during the test period under the influence of a temperature gradient along the length of the crucible. In the early tests, the temperature difference between the upper end of the capsule and the center was $15-20^{\circ}\text{C}$, and that between the bottom and the center was $20^{\circ}-25^{\circ}\text{C}$. By careful trimming of the heating element to provide an incremental heating at the top and bottom, the temperature differences have been reduced to about 5°C . The tendency here is for the metal to collect at the coldest part of the capsule. It is therefore advantageous if the crucible end of the capsule runs a few degrees cooler than the middle and the top of the capsule during the test period. However, it is difficult to determine the true temperature of the potassium surface in the crucible because the thermocouples contact the exterior of the capsule, and the internal gradient is probably different from that along the capsule wall. However, if the observed temperature difference is as large as 10°C , especially if the top of the capsule is the coolest region, and the seal between the crucible and capsule is poor, it does appear that a

significant amount of material could transfer and collect around the outside of the crucible. The reduction in the temperature differences along the capsule effected by trimming the heating element is therefore expected to have reduced markedly the potassium transfer by this mechanism, particularly when the top of the capsule runs slightly hotter than the bottom.

The third mechanism considered also involves vapor transfer, but is postulated to take place during the quenching step. During this period, the capsule wall is deliberately cooled. Since the heat transfer to and from the capsule-collector sub-assembly to the wall is relatively poor, the wall temperature could drop, say, 30-40°C before the collector and its collected potassium begins to cool. Under these circumstances, the potassium could readily vaporize, pass through a loose capsule-collector joint and condense on the cooled wall of the capsule. At present, this is thought to be the major mechanism contributing to the observed potassium transfer.

The solution of these transfer problems is not yet accomplished, although steps have been taken to control them. The first of these has been the reduction of the temperature gradient. The second involved an attempt to contain the potassium within the collector-crucible sub-assembly by trying to cause a pressure-weld to form between the collector and the crucible lip. The collector lip was cut with a 45° bevel, and the lips on the iron crucibles were machined to be square and true. Then the capsule was loaded using a spacer to ensure that pressure is applied to the joint, and with about a gram of potassium outside the crucible. This potassium provides an improved heat transfer bond between the capsule and collector, and also provides material to maintain the vapor pressure of potassium external to the crucible.

The improvement of the quenching action and the reduction of the time required to cool the capsule is another possible means of controlling the potassium transfer. Figure 2 shows a typical quenching curve of a capsule which is cooled from 1000°C and the corresponding temperature history of the chill blocks. The dashed curve represents a typical temperature history of the capsule wall. The collector, which presumably now contains the test potassium, is expected to follow a curve similar to the dashed curve shown in Figure 2 but which is displaced about 10-15 seconds to the right. Thus, in the first few seconds, a 30°-40°C temperature difference between the capsule

wall and the collector is developed. This difference decreases as the temperature level drops and is expected to be quite small by the time the capsule temperature has dropped to that of the chill block. With the present chill blocks, the time required for the capsule to cool to the melting point of potassium is four to six hours. Ideally, this time should be quite short, and a water-cooled quenching device is being designed to replace the chill blocks.

A summary of the test conditions and the results of the runs performed during the quarter is shown in Table 3. The results of the first two tests are discussed above. The second pair of tests, #317 and #318, were carried out using potassium from the second product batch. Run #317 had a zirconium getter tab in the collector, and run #318 had no getter. The amounts of iron dissolved in eight hour exposures showed a much smaller differential than that observed in the first tests. This is interpreted as confirming the lower oxygen level in the second batch of potassium.

Runs #320, #321, #322, #323, #325, and #327 were made using carefully sized spacers inside the capsule to hold the crucible-collector joints under slight compression during the runs. Further, in the last three of these runs, a flat strip of molybdenum was also placed inside the capsule to aid in maintaining the crucible and collector in axial alignment. In addition, the heater was trimmed in increments to attempt to reduce the temperature difference between the ends and the center of the capsule. Inspection of Table 3 shows that significant losses of potassium were experienced in all of the runs, even though the temperature difference was markedly reduced during the sequence of tests.

In an attempt to improve the seal between the collector and the capsule, runs #332-336 were carried out using a 45° bevel on the lip of the collector, and a very carefully machined flat seating surface on the crucibles. In these tests, the intent was to develop a pressure-induced diffusion bond seal during the test period. To this end, the spacers used in the assembly of the capsules were made long enough so that the end of the collector extended about 1/32 inch above the top of the capsule. Then the cap was pressed firmly against the collector and welded to maintain the pressure on the collector-crucible sub-assembly. The combination of pressure and the sharp edge on the collector was intended to lead to the development of a

diffusion bond and seal. Inspection of Table 3 shows that in one of the five tests a good seal was obtained. This yield is rather low, but the tendency to form a bond is obviously in evidence, so that the next series of tests is being prepared using a matched 10° taper on both the collector and the crucible.

Inspection of the observations made on the various runs reported in Table 3 tends to support the view that a major fraction of the potassium must transfer during the quench operation. If this is true, then the best value for the concentration of solute in potassium is found by computing the concentration based upon the solute found in the collector and the total amount of potassium loaded into the crucible. These values are shown in Table 3 and those marked with an asterisk are plotted in the log iron concentration vs $1/T$ plot shown in Figure 3. The data are, of course, tentative, primarily because of the uncertainty about the time at which the observed net potassium transfer occurred. The present data are compared in Figure 3 with related data. The most recent data are those of J. H. Swisher, and were reported at the Fall 1964 Meeting of the Electrochemical Society in Washington D. C.⁽¹⁾ Swisher reported a solubility curve for polycrystalline iron in potassium containing a nominal 20 ppm O, which curve gives 196 ppm as the equilibrium solubility at 800°C . The difference between Swisher's value and the present one is rather large, and part of the difference may be caused by our not having reached equilibrium in an eight-hour exposure. A second factor which would have an effect on the solubility is the difference in purity of the iron samples used. In this regard, the present metal, which was triple-pass zone-refined, would be expected to be purer, and to have a somewhat lower solubility. However, it is difficult to estimate the magnitude of these solubility-perturbing effects at this time.

It is also of some interest to compare the present values of the concentrations of our pure iron in potassium with the data of A. D. Bogard for the solubility of a very highly purified iron in sodium, as reported in NRL Report 4131.⁽²⁾ Bogard reports data taken at low temperatures (230° – 544°C) using a radioactive tracer analytical technique. Extrapolation of his data indicates a solubility level at 800°C of 3×10^{-6} w % or 0.03 wppm. The difference between this value and the present one cannot be readily explained.

Some of it must be due to the difference between sodium and potassium as solvents, to the difference in liquid metal purity, and to the difference in iron purity, but the question that remains is whether these differences do account for the solubility difference.

The lower solubility curve shown in Figure 3 was reported in the Sodium-NaK Supplement of the Liquid Metals Handbook.⁽³⁾ The data were developed by L. F. Epstein and cover the temperature range 225°-500°C. The values are lower than those found in the present program, and have a low temperature dependence, with an apparent $\Delta H(\text{solution})$ of 2560 cal/mol. On the other hand, Swisher's data show a $\Delta H(\text{solution})$ of 28,200 cal/mol. The trend of the estimated $\Delta H(\text{solution})$ from the present data is perhaps a few kcal/mol less than that of Swisher.⁽¹⁾ Further analysis of these tentative data is not warranted at this time.

Material Procurement, Preparation, and Characterization

One niobium and one molybdenum electrochemically machined crucible have been received from Sifco. The niobium crucible had been handled a great deal and showed many surface scratches and dents where the electrode leads were clamped down. There was a question regarding the effect that this mechanical damage to the crystal would have on its retaining its single crystal character when it was heated. In order to remove as much of the damaged material as possible, the crystal was chemically etched rather heavily to remove the deep scratches and dents. It was then electropolished and prepared for a DLC area measurement. However, during the area measurement, the electrode touched the inside of the crucible and the resulting arc burned a hole in the crucible wall about 1/4" from the closed end. Upon examination, it was found that the drilled hole was badly off-center at the bottom, and a rather large region in the vicinity of the penetration was paper thin. In order to recover the crucible as a high purity Nb crucible (but, of course, not single crystal), the closed end was cut off and a taper machined on it to form a plug. Then, a matching taper was cut in the original top-end of the crucible. The plug was inserted and the seal was attempted using the electron beam welder. The weld had a very small leak, which was closed by a subsequent weld. We now have a short, fully usable, high purity niobium crucible which probably approaches in quality a true single crystal crucible.

This crucible is being used until we receive delivery on the second niobium crucible from Sifco.

The molybdenum single crystal crucible meets specifications and is an excellent product. It has been electropolished and its surface area is being measured.

The remaining Nb, Mo, and Ta single crystals have been sent by Sifco to have undersize holes drilled in them using the Elox technique. Sifco expects to be able to clean up the Elox surface rather quickly with their ECM technique, and is expected to be able to deliver all the single crystal crucibles soon.

Chemical Analysis

During this quarter no further chemical analytical technique verification has been carried out. The activities have been limited to carrying out analyses of samples as required to support the experimental program. However, one interesting observation has been made with regard to the analysis of potassium for oxygen by the mercury amalgamation technique using the transfer vessels and closures. During the analytical procedure, the mercury is introduced into the vessel through a lightly-greased stopcock. In some of the analyses, the mercury would carry a few milligrams of the stopcock grease into the vessel. This grease tended to collect on the inner wall of the vessel and would usually trap a small amount of raw potassium there. The presence of grease was accompanied by the development of a dark coloration on the mercury amalgam surface. Apparently, the trapped potassium was not washed away in the normal sequence of mercury washing, so that it remained on the wall until the final aqueous washing step, at which time it would dissolve. Since there is no way of avoiding the grease in the vacuum stopcocks, the planned handling technique was abandoned.

The present sampling technique consists of dropping the potassium sample from the cutter wire directly into a small weighing bottle. Care is exercised to prevent the potassium's touching the lip of the bottle. The bottle is then closed using vacuum grease to seal its ground glass joint. The closed bottle is then removed from the vacuum system and placed in a scavenged-atmosphere argon dry box for analysis. The cap is removed, and the piece of potassium picked up on a stirring rod and placed in a separatory funnel with a greaseless telfon stopcock for analysis.

This technique has been quite successful, and was used in making the oxygen analyses shown in Table 1 and 2. The 16.2 ppm value shown was the average of seven analyses which ranged from 9 to 21 ppm. Four samples were taken shortly after the metal was prepared, and three samples were taken after most of the batch was used. The average analysis of the early samples was 15.9 ± 4.3 wppm O, that for the second set of samples was 16.7 ± 3.2 wppm O.

Table 1

Analyses of the First Batch of Potassium*
(wppm)

Element	Analysis	Element	Analysis	Element	Analysis
O	40-70	Cr	<1 D	Pb	ND
Ag	<1 ND	Cu	<2 D	Si	<5 D
Al	<2 ND	Fe	ND	Sn	ND
B	ND	Li	ND	Ti	ND
Ba	ND	Mg	<10 D	V	ND
Be	ND	Mn	ND	Zn	ND
Bi	ND	Mo	ND	Zr	ND
Ca	<1 D	Na	2 D	Nb	ND
Cd	ND	Ni	ND	Ta	ND
Co	ND				

*Spectroscopic analyses, except for O which was analyzed by the mercury amalgamation process, using the original transfer vessel technique.

Table 2

Analyses of the Second Batch of Potassium*
(wppm)

Element	Analysis	Element	Analysis	Element	Analysis
O	16.2 ± 3.9	Cd	<5 ND	Mo	<10 ND
C	<5	Co	<5 ND	Na	10
Ag	<1 D	Cr	<1 ND	Ni	<5 ND
Al	<5 D	Cu	<1 ND	Pb	<5 ND
Ba	<10 ND	Fe	<2 ND	Sn	<10 ND
Be	<1 ND	Li	<1 ND	Ti	<5 ND
Bi	<5 ND	Mg	<1 D	V	<5 ND
Ca	<1 D	Mn	<1 ND		

*Spectroscopic analyses, except for O (mercury amalgamation) and C, (wet oxidation). Analyses for B and Si are not shown because the sample was handled in quartz and showed evidence of Si and B contamination. The elements Zn, Zr, Nb, and Ta were not detected, but the spectroscopic sensitivity levels are high, being 500, 50, 50, and 50 wppm, respectively. Normally, when the analyses for these elements are of interest, they are run colorimetrically (See Report NASA-CR-54095, AI-64-235).

Table 3

Summary of Experimental Data on the Solution of Iron in Liquid Potassium

#	T °C	Time hr	Results	Temperature Differences, °C**		Comments
				Top	Bottom	
311*	800	2.6	25 ppm Fe	-	-	Analysis based on K in collector. Run terminated by vacuum leak; 99% of K in collector.
314*	800	2.6	15 ppm Fe	-	-	Analysis based on K in collector. Run scheduled to be equal in duration to #311; Zr getter in crucible; 76% of K in collector.
			11.4 ppm Fe			Analysis based on total K.
317	800	8.0	26 ppm Fe	-12	-10	Analysis based on K in collector; Zr getter in collector; 65% of K in collector.
			17 ppm Fe***			Analysis based on total K.
318	800	8.0	32 ppm Fe	-15	-11	Analysis based on K in collector; 45% of K in collector.
			14.5 ppm Fe***			Analysis based on total K.
320	800	4.0	38 ppm Fe	-14	-11	Analysis based on K in collector; Est. 55% of K in collector.
			(21 ppm Fe)***			Analysis based on estimated total K.
321	1000	4.0	-	-20	-17	No K in collector, no analysis.
322	1000	2.0	7 ppm Mo	-24	-26	Analysis based on K in collector; Est. 30% of K in collector. If K is lost only during quench step, the "true" Mo analysis would be about 2 ppm.
323	1000	4.0	-	-18	-16	Very little K in collector, no analysis.
325	600	8.0	-	0	-1	Very little K in collector, no analysis.

Table 3 (continued)

#	T °C	Time Hr	Results	Temperature Differences, °C**		Comments
				Top	Bottom	
326	1000	2.0	Mo	-8	-18	Sample not inverted after heating (to check inversion transfer loss), Est. 40% of K remained in crucible.
327	1000	2.0	82 ppm Fe	-4	-8	Analysis based on K in collector; 63% of K in collector.
			51.4 ppm Fe***			Analysis based on total K.
332	1000	4.0	269 ppm Fe	0	-8****	Analysis based on K in collector; 20% of K in collector, knife-edge.
			65.6 ppm Fe***			Analysis based on total K.
333	1000	4.0	74 ppm Fe	0	-6****	Analysis based on K in collector; 98% of K in collector; knife-edge; formed diffusion-bond seal.
			72 ppm Fe***			Analysis based on total K.
334	600	8.0	116 ppm Fe	0	-6****	Analysis based on K in collector; Est. 45% of K in collector; K in capsule; knife-edge.
			52.4 ppm Fe (?)			Analysis based on measured K loaded into crucible.
335	1000	4.0	(Mo)	0	-10	No K in collector.
336	1200	2.0	782 ppm Fe***	0	-15	Analysis based on K in collector. At temp. the volume of the K loaded exceeded volume of crucible, K in capsule, knife-edge; formed diffusion-bond seal.
337	1000	4.0	(Nb)	0	-5****	No K in collector.

*Runs using K from first batch (40-70 ppm O); all other runs use K from second batch (16 ppm O).

**Temperature differences are relative to mid-length temperature.

***Points plotted on Figure 1.

****These temperature profiles are considered to be very favorable.

FIGURE 1
TEST CAPSULE ASSEMBLY

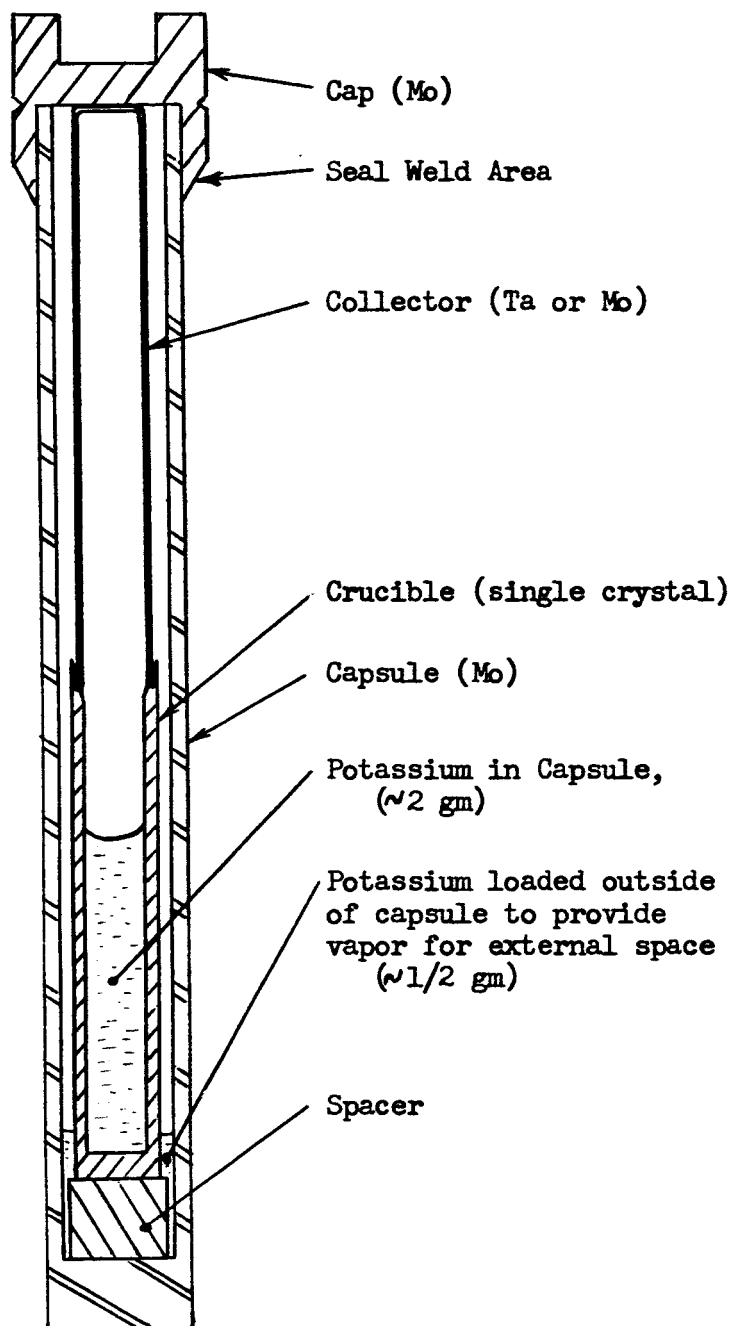


FIGURE 2
COOLING CURVES FOR QUENCHING USING CHILL BLOCKS

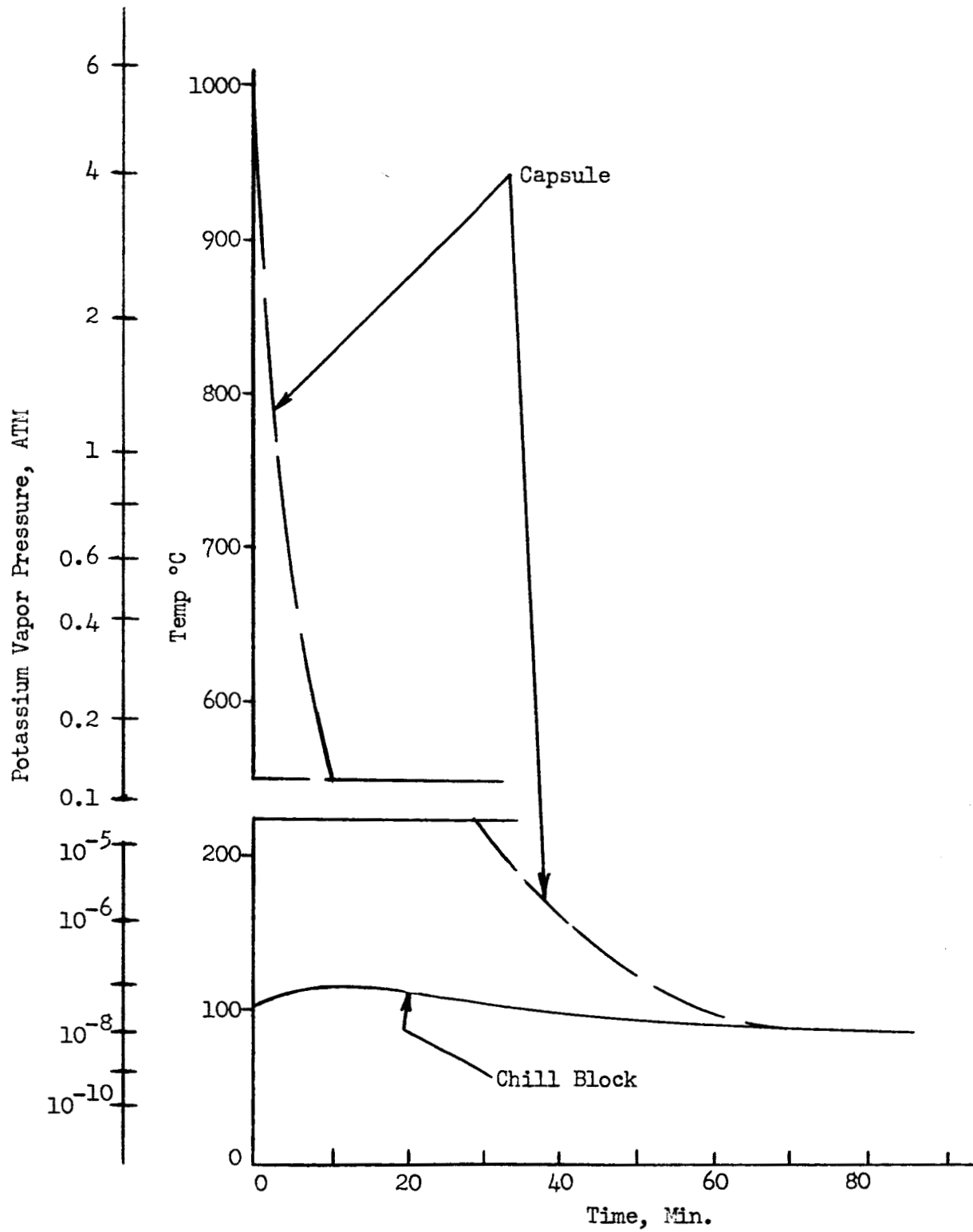
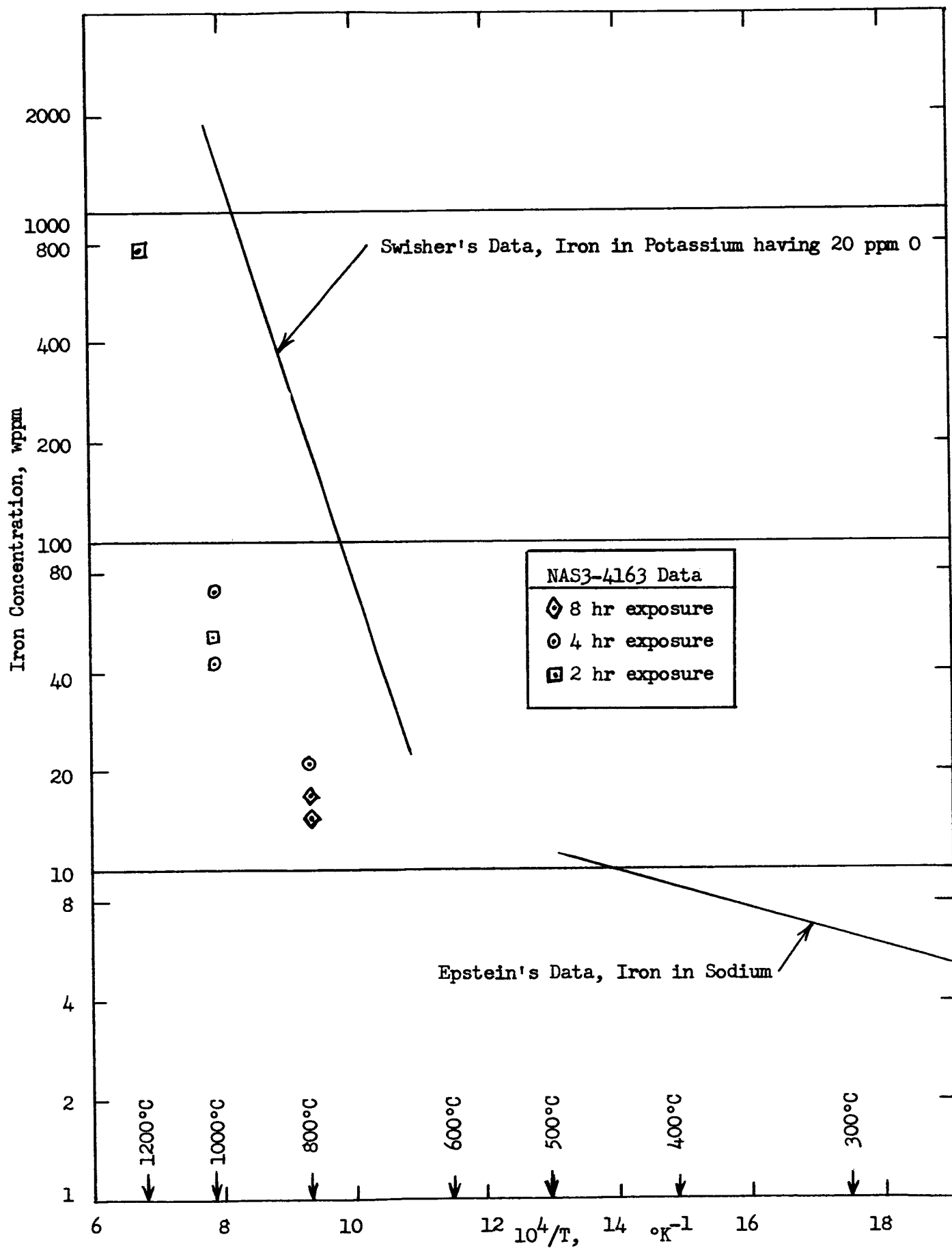


FIGURE 3

CONCENTRATION OF IRON DISSOLVED IN POTASSIUM CONTAINING 16 ppm O



IV. NEXT REPORT PERIOD ACTIVITIES

During the next quarter, the major effort will be directed toward solving the potassium transfer problem. Additional experimental measurements on the polycrystalline iron samples, on such single crystal materials as become available, and on the high-purity non-single-crystal molybdenum and niobium crucibles presently on hand, will be carried out after the transfer problem is solved.

V. PROJECT REPORTS ISSUED ON THIS CONTRACT

- R. L. McKisson, R. L. Eichelberger, and J. M. Scarborough,
"Solubility and Diffusion Studies of Ultra Pure Transition
Elements in Ultra Pure Alkali Metals," First Quarterly Report,
AI-9151, November 6, 1963.
- R. L. McKisson, R. L. Eichelberger, and G. R. Argue,
"Solubility and Diffusion Studies in Alkali Metals," Second
Quarterly Report, AI-64-5, February 7, 1964.
- R. L. McKisson, R. L. Eichelberger, G. R. Argue, and J. M. Scarborough,
"Solubility and Diffusion Studies in Alkali Metals," Third
Quarterly Report, NASA-CR-54043 (AI-64-75), May 11, 1964.
- R. L. McKisson, R. L. Eichelberger, G. R. Argue, and J. M. Scarborough,
"Solubility and Diffusion Studies in Alkali Metals," Fourth
Quarterly Report, NASA-CR-54094 (AI-64-156), August 10, 1964.
- R. L. McKisson and R. L. Eichelberger, "Solubility and Diffusion Studies
in Alkali Metals," Fifth Quarterly Report, NASA-CR-54095
(AI-64-235), November 16, 1964.

VI. AI INTERNAL REPORTS ISSUED ON THIS CONTRACT

- G. R. Argue, W. A. McCollum, and H. L. Recht, "Double Layer Capacitance Measurements on Ta, Mo, and Nb," AI-TDR-9773, March (1964).
- R. L. McKisson, "Analysis of the Freezing Point Depression Technique of Determining Impurity Content of Alkali Metal," AI-TDR-9404 (March, 1964).
- G. R. Argue, H. L. Recht, and W. A. McCollum, "Double Layer Capacitance Measurements on Iron Crucibles to Determine Surface Area," AI-TDR-64-229.

VII. REFERENCES

1. J. H. Swisher, "The Solubility of Iron and Nickel in Liquid Potassium from 1240° to 2000°F," paper presented at the Fall Meeting of the Electrochemical Society, Washington D. C., Oct. 11-15, 1964; also, Extended Abstracts of Electrothermics and Metallurgy Division, Volume 2, Number 2, page 13, Abstract No. 174.
2. A. D. Bogard, "The Solubility of Iron in Sodium Metal, Sodium-Sodium Oxide, and Sodium-Sodium Oxide-Sodium Hydroxide," NRL Report 4131, March 9, (1953).
3. Liquid Metals Handbook, Sodium-NaK Supplement, TID-5277, July 1, (1955), pg 16.